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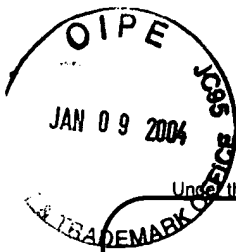


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PTO/SB/21 (08-03)

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TRANSMITTAL FORM (to be used for all correspondence after initial filing)	Application Number	10/091,062
	Filing Date	March 5, 2002
	First Named Inventor	Chen, et al.
	Art Unit	1616
	Examiner Name	Choi, Frank
Total Number of Pages in This Submission	Attorney Docket Number	366325-524

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January 7, 2004
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Attorney Docket No. 366325-524

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Chen, et al.

Serial No.: 10/091,062

Filed: March 5, 2002

For: Compositions and Methods for Mucosal Delivery

Group Art Unit: 1616

Examiner: Choi, Frank I.

Mail Stop: AF

Commissioner for Patents

P.O. Box 1450

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Communication

Sir:

Enclosed is a copy of the complete New York Times article (New York Times, Vol, 147, No. 51230, July 26, 1998, p. 4) for which only the abstract was previously provided with Information Disclosure Statement (Form PTO/SB/08B) originally filed on December 1, 2003 . Also enclosed is a copy of the 1995 ASTM D-1938-94 and D-882-94 standards requested by the Examiner.



Serial No.: 10091,062
Docket No.: 366325-524

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Respectfully submitted,

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An Upstart With Friends In the Highest of Places

All-Star Board Adds Cash, and Influence

By ANNE TERGESEN

ICOOS is anything but a typical development-stage biotechnology company.

Yes, like others in the field, it has yet to market a product or to make a profit from operations. (It announced on Thursday that it had lost \$9.4 million in the second quarter, or 24 cents a share, a bit more than analysts had forecast.) And a familiar kind of industry hype has begun to swirl around one of its projects — a compound that biotech analysts predict will supplant Pfizer's runaway hit, Imipramine drug, Viagra.

But look who's behind the Icos Corporation.

In a field populated by start-up operations run by starry-eyed scientists and littered with failures, Icos, based in Ballwin, Wis., has managed to attract some of the best of blue-chip names in corporate America to its board. Its roster of directors, which would make any Fortune 500 company proud, never mind a 75-employee fledgling, includes heavyweights like Walter B. Wriston, former chairman of Citicorp; Frank T. Cary, former president of LBM; James L. Fergeson, former chairman of General Foods; and William H. Gates, chairman of Microsoft.

Chalk up the A-list board to the credibility of Icos's chairman and chief executive, George B. Rathmann, whose previous effort in the field yielded the Amgen Corporation, the world's largest biotechnology company. "Obviously, his enormous success at Amgen gave anyone who was contemplating this thing a lot of confidence," said Mr. Wriston, who holds a stake in Icos valued at about \$5 million and — like the majority of the board — has been with the company since its inception in 1991.

Many of these luminaries have invested more than their time and reputations. Mr. Gates and Mr. Rathmann are Icos's two largest shareholders and have injected capital into the company several times. "If there was an equity offering, we would always be available," Mr. Rathmann said. Mr. Gates became involved in 1990, when Paley Webber was setting up Icos's first round of financing. "We had dinner, and the next day he put in \$5 million and joined our board," Mr. Rathmann said of Mr. Gates. Of course, having the country's richest man on the team is no guarantee of success. But it adds substance to the buzz surrounding Icos's patented Viagra replacement, a compound called IC351.

Too new to have a brand name, IC351

works in a way that is similar to Viagra and will be available in pill form, as Viagra is, but IC351 appears to be more selective than Viagra in blocking an enzyme that can prevent erections; the advantage may eliminate side effects associated with Viagra, including a temporary blue-green tinge in vision.

Madhava Murthy, senior biotechnology analyst at Lehman Brothers, said, "IC351 could be as large or larger than Viagra," which Lehman expects to generate \$30 million in sales this year and \$1.5 billion in 1997.

And analysts say Icos's drugs run a below-average risk of flopping in clinical trials because Icos does more careful

laboratory groundwork than many biotechnology bootstrappers. "Icos is very well known for the high quality of the research they do," said Mary Ann Gray, senior vice president at Raymond, James & Associates. "They take their time and do things correctly and have a solid background."

But Icos isn't depending on IC351 alone. In its research pipeline are a number of promising drugs aimed at large markets — a fact that cheers David Simpson, manager of the WM Northstar Fund and the WM Emerging Growth fund. The two funds bought Icos in 1995 and now have a \$13.4 million stake.

Icos is conducting preliminary trials of four drugs to treat nine diseases and conditions, including stroke, multiple sclerosis and asthma. Its goal is to have at least two of those compounds, possibly including IC351, in the pivotal third and final stage of clinical trials by the end of the year, said Larry J. Fitzpatrick, the company's assistant director for investor relations.

The great challenge faced by most biotechnology companies — how to keep the bills paid until products are ready to market — doesn't look like it will faze Icos. "Access to capital for our company is not going to be difficult, especially with a strong stock price and the board they have," said Ray Silverman, senior biotechnology analyst at Robertson Stephens. Most biotechnology companies find themselves having to trade away rights to their discoveries to raise develop-

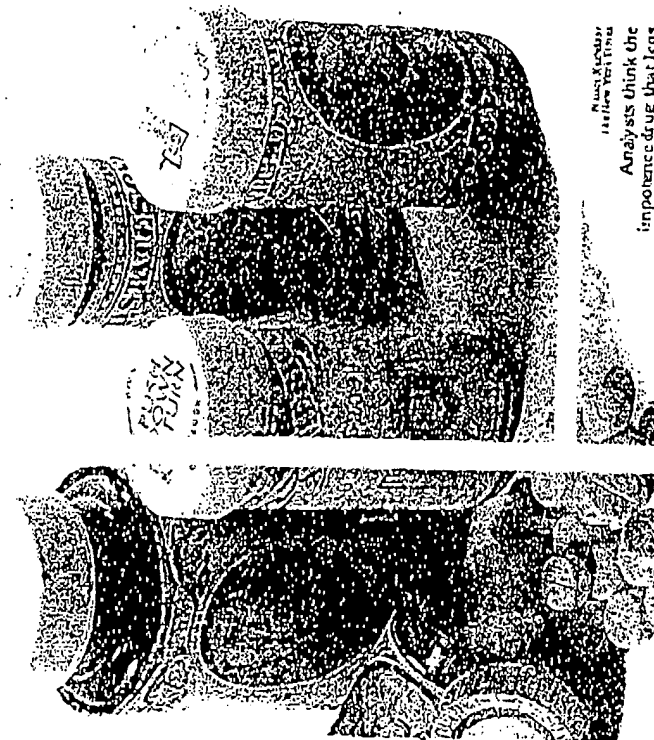


Photo by Andrew
Hoffman for Time

Analysts think the
impotence drug that Icos
is testing, IC351, has a chance

to supplant Pfizer's popular Viagra.
Other Icos drugs showing promise in
early tests include Leukarrest for circulatory
problems and multiple sclerosis, Palfase for asthma
and respiratory distress, and ICMi for psoriasis.

ment cash from richer partners, but Icos
can afford to hold tight to its rights and the
profits they promise, he added.

Last year Icos turned to creating limited
partnerships, a way of raising development
capital that fell out of favor in the biotech-
nology field in the early 1990's after several
product failures. The company has raised
about \$150 million so far by transferring
rights to three of its compounds (but not
IC351) to a partnership and selling interests
into. In addition to sharing in royalty income
from the drugs, the limited partners get
warrants to buy company stock; Icos re-
tains the right to buy back the partnership
interests. Mr. Gates and Mr. Rathmann
have each bought partnership stakes.

"The degree to which we control our
destiny is more important than anything
else," Mr. Rathmann said.

Mr. Simpson, the fund manager, is im-
pressed with Icos's ability to retain its inde-
pendence. "If they chose to partner with one
of the big pharmaceutical companies, they
don't have to give away all the value in
order to get money for research," he ob-
served. Icos has forged alliances with Ab-
bott Laboratories and Japan's Sumitomo, un-

der which Icos shares but does not surren-
der rights. Analysts expect something simi-
lar for IC351.

Given Icos's advantages is the compa-
ny's stock price, now \$12 1/4 a good value?
A growing number of short-sellers appar-
ently believe the stock is headed for a fall.
As of June 15, the uncovered short interest
in Icos stock had swollen to 2.3 million
shares, triple the level of a month earlier.

But Mr. Simpson disagreed with the
short-sellers. "I don't think it's overvalued,"
he said. "It's tough to find biotechnology
companies with true drugs in phase 2 trials.
If they all flop, you are overpaying, but if
just one of them works, you're well reward-
ed."

Analysts say investors in the volatile bio-
technology field must take the long view. "I
would never recommend someone buy the
stock unless they were long-term holders,
by which I mean at least three years," said
Andrew Hayward, director of research at
Rosenblatt Securities.

By next time Icos expects to have at least
one product on the market — all it should
need to turn a profit, Mr. Rathmann
said.



Standard Test Method for Tear-Propagation Resistance of Plastic Film and Thin Sheeting by a Single-Tear Method¹

This standard is issued under the fixed designation D 1938; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the determination of the force necessary to propagate a tear in plastic film and thin sheeting (thickness of 1 mm (0.04 in.) or less) by a single-tear method.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—This standard and ISO 6383-1 are technically equivalent. However, the specimen size is larger for ISO 6383-1.

2. Referenced Documents

2.1 ASTM Standards:

D 374 Test Methods for Thickness of Solid Electrical Insulation²

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing³

D 882 Test Methods for Tensile Properties of Thin Plastic Sheet³

D 883 Terminology Relating to Plastics³

D 4000 Classification System for Specifying Plastic Materials⁴

E 4 Practices for Load Verification of Testing Machines⁵

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁶

2.2 ISO Standard:

ISO 6383-1 Film and Sheet—Determination of Tear Resistance Part 1 Trousers Tear Method⁷

3. Terminology

3.1 **Definitions**—Definitions of terms applying to this test method appear in Terminology D 883.

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

Current edition approved Feb. 15, 1994. Published April 1994. Originally published as D 1938 - 62 T. Last previous edition D 1938 - 93.

² Annual Book of ASTM Standards, Vol 10.01.

³ Annual Book of ASTM Standards, Vol 08.01.

⁴ Annual Book of ASTM Standards, Vol 08.02.

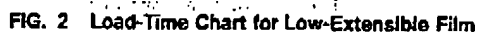
⁵ Annual Book of ASTM Standards, Vol 03.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

TABLE 1 Tear Propagation Resistance (Tear Force) Values
Tear Force Values Expressed in Units of Grams Force

Thickness, mm (in.)	50 mm (2 in.)	75 mm (3 in.)
0.04 (0.0016)	1000 (22.0)	1500 (33.0)
0.05 (0.0020)	1200 (26.4)	1800 (40.0)
0.06 (0.0024)	1400 (30.9)	2100 (46.2)
0.08 (0.0031)	1800 (40.0)	2700 (60.0)
0.10 (0.0039)	2200 (49.1)	3300 (73.4)
0.12 (0.0047)	2600 (57.8)	3900 (86.9)
0.15 (0.0059)	3300 (73.4)	4950 (110.0)
0.20 (0.0079)	4400 (97.9)	6600 (146.3)
0.25 (0.0100)	5500 (122.5)	8250 (183.8)
0.30 (0.0118)	6600 (146.3)	9900 (219.9)
0.40 (0.0157)	8800 (195.8)	13200 (295.3)
0.50 (0.0197)	11000 (244.5)	16500 (367.9)
0.60 (0.0236)	13200 (295.3)	19800 (441.3)
0.80 (0.0315)	17600 (393.7)	26400 (588.8)
1.00 (0.0394)	22000 (491.3)	33000 (734.3)
1.20 (0.0472)	26400 (588.8)	39600 (879.9)
1.50 (0.0591)	33000 (734.3)	49500 (1100.0)
2.00 (0.0787)	44000 (978.9)	66000 (1463.0)
2.50 (0.0984)	55000 (1224.5)	82500 (1837.5)
3.00 (0.1181)	66000 (1469.1)	99000 (2199.0)
4.00 (0.1575)	88000 (1957.5)	132000 (2952.5)
5.00 (0.1968)	110000 (2444.8)	165000 (3676.8)
6.00 (0.2362)	132000 (2937.1)	198000 (4410.1)
8.00 (0.3150)	176000 (3936.5)	264000 (5887.5)
10.00 (0.3937)	220000 (4912.9)	330000 (7342.9)
12.00 (0.4724)	264000 (5888.3)	396000 (8797.3)
15.00 (0.5907)	330000 (7342.9)	495000 (11000.0)
20.00 (0.7874)	440000 (9789.1)	660000 (14630.0)
25.00 (0.9842)	550000 (12245.3)	825000 (18375.0)
30.00 (1.1811)	660000 (14691.5)	990000 (21990.0)
40.00 (1.5748)	880000 (19575.0)	1320000 (29525.0)
50.00 (1.9685)	1100000 (24447.9)	1650000 (36767.9)
60.00 (2.3622)	1320000 (29371.4)	1980000 (44101.4)
80.00 (3.1496)	1760000 (39364.5)	2640000 (58875.0)
100.00 (3.9370)	2200000 (49129.1)	3300000 (73429.1)
120.00 (4.7244)	2640000 (58883.3)	3960000 (87973.3)
150.00 (5.9072)	3300000 (73429.1)	4950000 (110000.0)
200.00 (7.8740)	4400000 (97891.3)	6600000 (146300.0)
250.00 (9.8425)	5500000 (122452.5)	8250000 (183750.0)
300.00 (11.8110)	6600000 (146913.7)	9900000 (219900.0)
400.00 (15.7480)	8800000 (195750.0)	13200000 (295250.0)
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600.00 (23.6220)	13200000 (293714.3)	19800000 (441014.3)
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8000000000.00 (31.4960)	176000000000000 (3936452500000.0)	264000000000000 (5887500000000.0)
10000000000.00 (39.3700)	220000000000000 (4912909090909.1)	330000000000000 (7342909090909.1)
12000000000.00 (47.2440)	264000000000000 (5888327272727.3)	396000000000000 (8797327272727.3)
15000000000.00 (5.9072)	330000000000000 (7342909090909.1)	495000000000000 (11000000000000.0)
20000000000.00 (7.8740)	440000000000000 (9789136500000.0)	660000000000000 (14630000000000.0)
25000000000.00 (9.8425)	550000000000000 (12245250000000.0)	825000000000000 (18375000000000.0)
30000000000.00 (11.8110)	660000000000000 (14691365000000.0)	990000000000000 (21990000000000.0)
40000000000.00 (15.7480)	880000000000000 (19575000000000.0)	1320000000000000 (29525000000000.0)
50000000000.00 (19.6850)	1100000000000000 (24447909090909.1)	1650000000000000 (36767909090909.1)
60000000000.00 (23.6220)	1320000000000000 (29371429090909.1)	1980000000000000 (44101429090909.1)
80000000000.00 (31.4960)	1760000000000000 (39364525000000.0)	2640000000000000 (58875000000000.0)
100000000000.00 (39.3700)	2200000000000000 (49129090909090.9)	3300000000000000 (73429090909090.9)
120000000000.00 (47.2440)	2640000000000000 (58883272727273.0)	3960000000000000 (87973272727273.0)
150000000000.00 (5.9072)	3300000000000000 (73429090909091.0)	4950000000000000 (110000000000000.0)
200000000000.00 (7.8740)	4400000000000000 (97891365000000.0)	6600000000000000 (146300000000000.0)
250000000000.00 (9.8425)	5500000000000000 (122452500000000.0)	8250000000000000 (183750000000000.0)
300000000000.00 (11.8110)	6600000000000000 (146913650000000.0)	9900000000000000 (219900000000000.0)
400000000000.00 (15.7480)	8800000000000000 (195750000000000.0)	13200000000000000 (295250000000000.0)
500000000000.00 (19.6850)	11000000000000000 (244479090909090.9)	16500000000000000 (367679090909090.9)
600000000000.00 (23.6220)	13200000000000000 (293714290909090.9)	19800000000000000 (441014290909090.9)
800000000000.00 (31.4960)	17600000000000000 (393645250000000.0)	26400000000000000 (588750000000000.0)
1000000000000.00 (39.3700)	22000000000000000 (491290909090909.1)	33000000000000000 (734290909090909.1)
1200000000000.00 (47.2440)	26400000000000000 (588832727272727.3)	39600000000000000 (879732727272727.3)
1500000000000.00 (5.9072)	33000000000000000 (734290909090909.1)	49500000000000000 (1100000000000000.0)
2000000000000.00 (7.8740)	44000000000000000 (978913650000000.0)	66000000000000000 (1463000000000000.0)
2500000000000.00 (9.8425)	55000000000000000 (1224525000000000.0)	82500000000000000 (1837500000000000.0)
3000000000000.00 (11.8110)	66000000000000000 (1469136500000000.0)	99000000000000000 (2199000000000000.0)
4000000000000.00 (15.7480)	88000000000000000 (1957500000000000.0)	132000000000000000 (2952500000000000.0)
5000000000000.00 (19.6850)	110000000000000000 (2444790909090909.1)	165000000000000000 (3676790909090909.1)



7.3 Cut enough specimens to provide a minimum of five tear-propagation force determinations each in the machine direction and in the transverse direction of the material being tested.

8.2 Test Conditions—Conduct tests in the standard laboratory atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity, unless otherwise specified in the test methods. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and $\pm 2\%$ relative humidity.

9.3 Test not less than five specimens in each of the principal film or sheeting directions.

10.4 Calculate the estimated standard deviation as follows and report to the significant figures:

$$s = \sqrt{(\sum X^2 - n\bar{X}^2)/(\eta - 1)}$$

s = estimated standard deviation,
 X = value of a single observation,
 n = number of observations, and
 \bar{X} = arithmetic means of the set of observations.

11. Report

11.1 Report the following information:

11.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, orientation of samples with respect to principal directions of the material, etc.

11.1.2 Average thickness of test specimens,

11.1.3 Number of samples tested,

11.1.4 Date of test, and

11.1.5 Mean of the five average tear-propagation determinations, usually in newtons (or pounds-force), for the materials described in 10.1; and the mean of the five initial tear-propagation forces and the mean of the five maximum tear-propagation forces, usually in newtons (or pounds-force), for materials described in 10.2. In each case, report the standard deviation of the above sets of data. In the cases where the specimens tear to one side, so state, and report the values obtained.

12. Precision and Bias

12.1 Precision:

12.1.1 Tables 1 and 2 are based on a round robin⁸ conducted between 1986 and 1990 in accordance with Practice E 691-87, involving seven materials tested by seven laboratories. For each material, all the samples were prepared at one source, and randomized sections of film were sent to each of the laboratories which prepared the test specimens and tested them. Each test result was the average of five determinations. Each laboratory obtained two test results for each material.

NOTE 3: Caution—The following explanations of r and R (12.1.2 through 12.1.2.3) are intended only to present a meaningful way of considering the approximate precision of this test method. The data in Tables 1 and 2 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691-87 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 12.1.2 through 12.1.2.3 would then be valid for such data.

12.1.2 Concept of r and R —If S_r and S_R have been calculated from a large enough body of data and for test results that were the result of testing five specimens, the following applies:

12.1.2.1 Repeatability Limit, r —In comparing two test results for the same material obtained by the same operator using the same equipment on the same day, the two test

TABLE 1 Tear Propagation Resistance (Trouser Tear) Machine Direction (Values Expressed in Units of Grams-Force)

Material	Average	S_r^A	S_R^B	r^C	R^D
Polystyrene	5.04	1.54	3.47	4.32	9.72
Polyester	182.5	108.1	172.0	19.81	19.81
Polypropylene	70.77	20.52	38.05	57.45	108.6
HDPE No. 2	146.04	46.04	115.56	134.59	158.2
LDPE—LD 104	228.3	33.98	33.98	95.14	95.14
LLDPE	337.1	30.95	42.74	88.88	119.7
HDPE No. 1	482.9	49.04	108.0	137.3	296.9

^A S_r = within-laboratory standard deviation for the material stated. It is obtained by pooling the standard deviations of the test results from each laboratory, as follows:

$$S_r = [(S_1)^2 + (S_2)^2 + \dots + (S_n)^2 / n]^{1/2}$$

^B S_R = between-laboratories standard deviation for the material stated. It is a pooling of the amounts by which the average of the test results for each laboratory deviate from the overall average for that material.

^C r = within-laboratory repeatability limit = $2.8 \times S_r$

^D R = between-laboratories reproducibility limit = $2.8 \times S_R$

TABLE 2 Tear Propagation Resistance (Trouser Tear) Transverse Direction (Values Expressed in Units of Grams-Force)

Material	Average	S_r^A	S_R^B	r^C	R^D
Polystyrene	3.86	0.46	3.06	1.28	8.63
Polyester	32.47	1.74	3.68	4.88	10.31
LDPE—LD 104	278.6	12.21	30.29	34.18	84.40
Polypropylene	326.2	49.67	124.9	139.1	349.7
LLDPE	372.5	26.89	31.68	74.74	88.70
HDPE No. 2	452.6	24.68	31.28	69.10	87.59
HDPE No. 1	549.7	64.10	105.4	179.5	295.0

^A S_r = within-laboratory standard deviation for the material stated. It is obtained by pooling the standard deviations of the test results from each laboratory, as follows:

$$S_r = [(S_1)^2 + (S_2)^2 + \dots + (S_n)^2 / n]^{1/2}$$

^B S_R = between-laboratories standard deviation for the material stated. It is a pooling of the amounts by which the average of the test results for each laboratory deviate from the overall average for that material.

^C r = within-laboratory repeatability limit = $2.8 \times S_r$

^D R = between-laboratories reproducibility limit = $2.8 \times S_R$

results should be judged not equivalent if they differ by more than the r value for that material.

12.1.2.2 Reproducibility Limit, R —In comparing two test results for the same material obtained by different operators using different equipment in different laboratories, the two test results should be judged not equivalent if they differ by more than the R value for that material.

12.1.2.3 Any judgment in accordance with 12.1.2.1 or 12.1.2.2 would have an approximate 95 % (0.95) probability of being correct.

12.2 Bias—There are no recognized standards to estimate the bias of this test method.

13. Keywords

13.1 plastic film; single tear; tear; thin sheeting; trouser

⁸ Supporting data on precision are available from ASTM Headquarters. Request RR: D20-1177.

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Standard Test Methods for Tensile Properties of Thin Plastic Sheeting¹

This standard is issued under the fixed designation D 882; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 1013 of Federal Test Method Standard 406. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of tensile properties of plastics in the form of thin sheeting, including film (less than 1.0 mm (0.04 in.) in thickness).

NOTE 1—Film has been arbitrarily defined as sheeting having nominal thickness not greater than 0.25 mm (0.010 in.).

NOTE 2—Tensile properties of plastics 1.0 mm (0.04 in.) or greater in thickness shall be determined according to Test Method D 638.

1.2 Two types of tension tests are described in these test methods, differing basically only in manner of load application. These test methods may be used to test all plastics within the thickness range described and the capacity of the machine employed.

1.2.1 *Test Method A: Static Weighing, Constant-Rate-of-Grip Separation Test*—This test method employs a constant rate of separation of the grips holding the ends of the test specimen.

1.2.2 *Test Method B: Pendulum Weighing, Constant-Rate-of-Power-Grip Motion Test*—This test method employs a constant rate of motion of one grip and a variable rate of motion of the second grip. The variable-rate grip is attached to a pendulum weighing head, and its movement is dependent on the load-deformation behavior of the material under test.

1.3 Specimen extension may be measured in these test methods by grip separation, extension indicators, or displacement of gage marks.

1.4 A procedure for determining the tensile modulus of elasticity is included, using Test Method A at one strain rate.

1.5 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information only.

NOTE 3—This modulus determination procedure is based on the use of grip separation as a measure of extension; however, the desirability of using extension indicators accurate to $\pm 1.0\%$ or better as specified in Test Method D 638 is recognized, and provision for the use of such instrumentation is incorporated in the procedure.

1.6 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. A specific

precautionary statement is given in Note 19.

2. Referenced Documents

2.1 ASTM Standards:

- D 374 Test Methods for Thickness of Solid Electrical Insulation²
- D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing³
- D 638 Test Method for Tensile Properties of Plastics³
- D 4000 Classification System for Specifying Plastic Materials⁴
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁵

3. Terminology

3.1 *Definitions*—Definitions of terms and symbols relating to tension testing of plastics appear in the Annex to Test Method D 638.

3.1.1 *line grips*—grips having faces designed to concentrate the entire gripping force along a single line perpendicular to the direction of testing stress. This is usually done by combining one standard flat face and an opposing face from which protrudes a half-round.

3.1.2 *tear failure*—a tensile failure characterized by fracture initiating at one edge of the specimen and progressing across the specimen at a rate slow enough to produce an anomalous load-deformation curve.

4. Significance and Use

4.1 Tensile properties determined by these test methods are of value for the identification and characterization of materials for control and specification purposes. Tensile properties may vary with specimen thickness, method of preparation, speed of testing, type of grips used, and manner of measuring extension. Consequently, where precise comparative results are desired, these factors must be carefully controlled. Since the actual loading rates vary between Test Methods A and B, the results obtained using these two methods cannot be directly compared. Test Method A is preferred and shall be used for referee purposes, unless otherwise indicated in particular material specifications. For many materials, there may be a specification that requires the use of this test method, but with some procedural

¹ These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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² Annual Book of ASTM Standards, Vol 10.01.

³ Annual Book of ASTM Standards, Vol 08.01.

⁴ Annual Book of ASTM Standards, Vol 08.02.

⁵ Annual Book of ASTM Standards, Vol 14.02.

modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 in Classification D 4000 lists the ASTM materials standards that currently exist.

4.2 Tensile properties may be utilized to provide data for research and development and engineering design as well as quality control and specification. However, data from such tests cannot be considered significant for applications differing widely from the load-time scale of the test employed.

4.3 The tensile modulus of elasticity is an index of the stiffness of thin plastic sheeting. The reproducibility of test results is good when precise control is maintained over all test conditions. When different materials are being compared for stiffness, specimens of identical dimensions must be employed.

4.4 The tensile energy to break (TEB) is the total energy absorbed per unit volume of the specimen up to the point of rupture. In some texts this property has been referred to as *toughness*. It is used to evaluate materials that may be subjected to heavy abuse or that might stall web transport equipment in the event of a machine malfunction in end-use applications. However, the rate of strain, specimen parameters, and especially flaws may cause large variations in the results. In that sense, caution is advised in utilizing TEB test results for end-use design applications.

4.5 Materials that fail by tearing give anomalous data which cannot be compared with those from normal failure.

5. Apparatus

5.1 *Grips*—A gripping system that minimizes both slippage and uneven stress distribution.

NOTE 4—Grips lined with thin rubber, crocus-cloth, or pressure-sensitive tape as well as file-faced or serrated grips have been successfully used for many materials. The choice of grip surface will depend on the material tested, thickness, etc. More recently, line grips padded on the round face with 1.0-mm (40-mil) blotting paper have been found superior. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to “neck” into the grips, since pressure is maintained at all times. In cases where samples frequently fail at the edge of the grips, it may be advantageous to increase slightly the radius of curvature of the edges where the grips come in contact with the test area of the specimen.

5.2 *Thickness Gage*—A dead-weight dial micrometer as prescribed in Method C of Test Methods D 374, reading to 0.0025 mm (0.0001 in.) or less.

5.3 *Width-Measuring Devices*—Suitable test scales or other width measuring devices capable of measuring 0.25 mm (0.010 in.) or less.

5.4 *Specimen Cutter*—Razor blades, fixtures incorporating razor blades, suitable paper cutters, or other devices capable of cutting the specimens to the proper width and producing straight, clean, parallel edges with no visible imperfections, shall be used. Devices that use razor blades have proved especially suitable for materials having an elongation-at-fracture above 10 to 20 %. A device consisting of two parallel knives mounted firmly against a precision-ground base shear block (similar to a paper cutter) has also proved satisfactory. The use of striking dies is not recommended because of poor and inconsistent specimen edges which may be produced. It is imperative that the cutting edges be kept sharp and free from visible scratches or nicks.

5.5 *Extension Indicators* (if employed), shall conform to requirements specified in Test Method D 638. In addition, such apparatus shall be so designed as to minimize stress on the specimen at the contact points of the specimen and the indicator (see 8.3).

5.6 *Testing Machines*—

5.6.1 *For Test Method A*—A testing machine of the constant rate-of-jaw-separation type. The machine shall be equipped with a weighing system that moves a maximum distance of 2 % of the specimen extension within the range being measured. The machine shall be equipped with a device for recording the tensile load and the amount of separation of the grips; both of these measuring systems shall be accurate to ± 2 %. The rate of separation of the jaws shall be uniform and capable of adjustment from approximately 1.3 to 500 mm (0.05 to 20 in.)/min in increments necessary to produce the strain rates specified in 9.3 and 9.4. This test method (A) shall be used for tensile modulus of elasticity measurements (Note 5).

5.6.2 *For Test Method B*—A testing machine of the pendulum type. This machine shall be equipped with a pendulum weighing head to measure the load applied to the test specimen and a device for indicating or recording the tensile load carried by the specimen with an accuracy of ± 2 %. The rate of travel of the power-activated grip shall be uniform and capable of adjustment to 50.8 and 508 mm (2 and 20 in.)/min.

NOTE 5—A high response speed in the recording system is desirable, particularly when relatively high strain rates are employed for rigid materials. The speed of pen response for recorders is supplied by manufacturers of this equipment. Care must be taken to conduct tests at conditions such that response time (ability of recorder to follow actual load) will produce less than 2 % error.

6. Test Specimens

6.1 The test specimens shall consist of strips of uniform width and thickness at least 50 mm (2 in.) longer than the grip separation used.

6.2 The nominal width of the specimens shall be not less than 5.0 mm (0.20 in.) or greater than 25.4 mm (1.0 in.).

6.3 A width-thickness ratio of at least eight shall be used. Narrow specimens magnify effects of edge strains or flaws, or both.

6.4 The utmost care shall be exercised in cutting specimens to prevent nicks and tears which are likely to cause premature failures (Note 6). The edges shall be parallel to within 5 % of the width over the length of the specimen between the grips.

NOTE 6—Microscopical examination of specimens may be used to detect flaws due to sample or specimen preparation.

6.5 Wherever possible, the test specimens shall be selected so that thickness is uniform to within 10 % of the thickness over the length of the specimen between the grips in the case of materials 0.25 mm (0.010 in.) or less in thickness and to within 5 % in the case of materials greater than 0.25 mm (0.010 in.) in thickness but less than 1.00 mm (0.040 in.) in thickness.

NOTE 7—In cases where thickness variations are in excess of those recommended in 6.5, results may not be characteristic of the material under test.

6.6 If the material is suspected of being anisotropic, two

sets of test specimens shall be prepared having their long axes respectively parallel with and normal to the suspected direction of anisotropy. For tensile modulus of elasticity determinations, a specimen gage length of 250 mm (10 in.) shall be considered as standard. This length is used in order to minimize the effects of grip slippage on test results. When this length is not feasible, test sections as short as 100 mm (4 in.) may be used if it has been shown that results are not appreciably affected. However, the 250-mm gage length shall be used for referee purposes. The speed of testing of shorter specimens must be adjusted in order for the strain rate to be equivalent to that of the standard specimen.

NOTE 8—Two round robin tests⁶ have shown that, for materials of less than 0.25-mm (10-mil) thickness, line grips padded on the round side with 1.0-mm (40-mil) blotting paper give the same results with a 100-mm test section as a 250-mm test section produces with flat-face grips.

NOTE 9—Excessive jaw slippage becomes increasingly difficult to overcome in cases where high modulus materials are tested in thicknesses greater than 0.25 mm (0.010 in.).

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 for those tests where conditioning is required. In cases of disagreement, the tolerances shall be 1°C (1.8°F) and $\pm 2\%$ relative humidity.

7.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) and $50 \pm 5\%$ relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreement, the tolerances shall be $\pm 1^\circ\text{C}$ ($\pm 1.8^\circ\text{F}$) and $\pm 2\%$ relative humidity.

8. Number of Test Specimens

8.1 In the case of isotropic materials, at least five specimens shall be tested from each sample.

8.2 In the case of anisotropic materials, at least ten specimens, five normal and five parallel with the principal axis of anisotropy, shall be tested from each sample.

8.3 Specimens that fail at some obvious flaw or that fail outside the gage length shall be discarded and retests made, unless such flaws or conditions constitute a variable whose effect is being studied. However, jaw breaks (failures at the grip contact point) are acceptable if it has been shown that results from such tests are in essential agreement with values obtained from breaks occurring within the gage length.

NOTE 10—In the case of some materials, examination of specimens, prior to and following testing, under crossed optical polarizers (polarizing films) provides a useful means of detecting flaws which may be, or are, responsible for premature failure.

9. Speed of Testing

9.1 The speed of testing is the rate of separation of the two members (or grips) of the testing machine when running idle (under no load). This rate of separation shall be maintained

within 5% of the no-load value when running under full-capacity load. The speed of testing shall be calculated from the required initial strain rate as specified in Table 1. The rate of grip separation may be determined for the purpose of these test methods from the initial strain rate as follows:

where:

A = rate of grip separation, mm (or in.)/min, and
 B = initial distance between grips, mm (or in.), and
 C = initial strain rate, mm/mm·min (or in./in.·min).

9.3 The initial strain rate shall be as in Table 1 unless otherwise indicated by the specification for the material being tested.

NOTE 11—Results obtained at different initial strain rates are not comparable; consequently, where direct comparisons between materials in various elongation classes are required, a single initial strain rate should be used. For some materials it may be advisable to select the strain rates on the basis of percent elongation at yield.

9.4 In cases where conflicting material classification, as determined by percent elongation at break values, results in a choice of strain rates, the lower rate shall be used.

9.5 If modulus values are being determined, separate specimens shall be used whenever strain rates and specimen dimensions are not the same as those employed in the test for other tensile properties.

10. Procedure

10.1 Select a load range such that specimen failure occurs within its upper two thirds. A few trial runs may be necessary to select a proper combination of load range and specimen width.

10.2 Measure the cross-sectional area of the specimen at several points along its length. Measure the width to an accuracy of 0.25 mm (0.010 in.) or better. Measure the thickness to an accuracy of 0.0025 mm (0.0001 in.) or better for films less than 0.25 mm (0.010 in.) in thickness and to an accuracy of 1% or better for films greater than 0.25 mm (0.010 in.) but less than 1.0 mm (0.040 in.) in thickness.

10.3 The initial grip separation shall be at least 50 mm (2 in.) for materials having a total elongation at break of 100% or more, and at least 100 mm (4 in.) for materials having a total elongation at break of less than 100%.

NOTE 12—Since slippage is a potential problem in these tests, as great an initial distance between grips as possible should be employed.

10.4 Set the rate of grip separation to give the desired strain rate based on the initial distance between the grips

TABLE 1 Speed of Testing

Test Method	Percent Elongation at Break	Initial Strain Rate, mm/mm·min (in./in.·min)
Modulus of Elasticity Determination		
A		0.1
Determinations other than Elastic Modulus		
A	Less than 20	0.1
	20 to 100	0.5
	Greater than 100	10.0
B	Less than 100	0.5
	Greater than 100	10.0

⁶ Supporting data are available from ASTM Headquarters. Request RR: D20-1038.

Note 13. Balance, zero, and calibrate the load weighing and recording system.

Note 13—Suggested crosshead speeds and initial grip separation to give the desired initial strain rate, described in Table 1 are shown in Table 2.

10.5 In cases where it is desired to measure a test section other than the total length between the grips, mark the ends of the desired test section with a soft, fine wax crayon or with ink. Do not scratch these marks onto the surface since such scratches may act as stress raisers and cause premature specimen failure. Extensometers may be used if available; in this case, the test section will be defined by the contact points of the extensometer.

Note 14—Measurement of a specific test section is necessary with some materials having high elongation. As the specimen elongates, the accompanying reduction in area results in a loosening of material at the inside edge of the grips. This reduction and loosening moves back into the grips as further elongation and reduction in area takes place. In effect, this causes problems similar to grip slippage, that is, exaggerates measured extension.

10.6 Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize slipping of the specimen during test.

10.7 Start the machine and record load versus extension.

10.7.1 When the total length between the grips is used as the test area, record load versus grip separation.

10.7.2 When a specific test area has been marked on the specimen, follow the displacement of the edge boundary lines with respect to each other with dividers or some other suitable device. If a load-extension curve is desired, plot various extensions versus corresponding loads sustained, as measured by the load indicator.

10.7.3 When an extensometer is used, record load versus extension of the test area measured by the extensometer.

10.8 If modulus values are being determined, select a load range and chart rate to produce a load-extension curve of between 30 and 60° to the X axis. For maximum accuracy, use the most sensitive load scale for which this condition can be met. The test may be discontinued when the load-extension curve deviates from linearity.

10.9 In the case of materials being evaluated for secant modulus, the test may be discontinued when the specified extension has been reached.

10.10 If tensile energy to break is being determined, some provision must be made for integration of the stress-strain

curve. This may be either an electronic integration during the test or a subsequent determination from the area of the finished stress-strain curve (see Annex A2).

11. Calculation

11.1 **Breaking Factor** (nominal) shall be calculated by dividing the maximum load by the original minimum width of the specimen. The result shall be expressed in force per unit of width, usually newtons per metre (or pounds per inch) of width, and reported to three significant figures. The thickness of the film shall always be stated to the nearest 0.0025 mm (0.0001 in.).

Example—Breaking Factor = 1.75 kN/m (10.0 lbf/in.) of width for 0.1300-mm (0.0051-in.) thickness.

Note 15—This method of reporting is useful for very thin films (0.13 mm (0.005 in.) and less) for which breaking load may not be proportional to cross-sectional area and whose thickness may be difficult to determine with precision. Furthermore, films which are in effect laminar due to orientation, skin effects, nonuniform crystallinity, etc., have tensile properties disproportionate to cross-sectional area.

11.2 **Tensile Strength** (nominal) shall be calculated by dividing the maximum load by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures.

Note 16—When tear failure occurs, so indicate and calculate results based on load and elongation at which tear initiates, as reflected in the load-deformation curve.

11.3 **Tensile Strength at Break** (nominal) shall be calculated in the same way as the tensile strength except that the load at break shall be used in place of the maximum load (Notes 16 and 17).

Note 17—In many cases tensile strength and tensile strength at break are identical.

11.4 **Percent Elongation at Break** shall be calculated by dividing the extension at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation; otherwise the distance between the grips shall be used. The result shall be expressed in percent and reported to two significant figures (Note 16).

11.5 **Yield Strength**, where applicable, shall be calculated by dividing the load at the yield point by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be

TABLE 2 Crosshead Speeds and Initial Grip Separation

Test Method	Percent Elongation at Break	Initial Strain Rate,	Initial Grip Separation		Rate of Grip Separation	
		mm/mm·min (in./in.·min)	mm	in.	mm/min	in./min
Modulus of Elasticity Determination						
A		0.1	250	10	25	1.0
Determinations other than Elastic Modulus						
A	Less than 20	0.1	125	5	12.5	0.5
	20 to 100	0.5	100	4	50	2.0
	Greater than 100	10.0	50	2	500	20.0
B	Less than 100	0.5	100	4	50	2.0
	Greater than 100	10.0	50	2	500	20.0

reported to three significant figures. Alternatively, for materials that exhibit Hookean behavior in the initial part of the curve, an offset yield strength may be obtained as described in the Appendix of Test Method D 638. In this case the value should be given as "yield strength at —% offset."

11.6 *Percent Elongation at Yield*, where applicable, shall be calculated by dividing the extension at the yield point by the initial gage length of specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation. Before calculating, correct the extension for "toe compensation" as described in Annex A1. The results shall be expressed in percent and reported to two significant figures. When offset yield strength is used, the elongation at the offset yield strength may be calculated.

11.7 *Elastic Modulus* shall be calculated by drawing a tangent to the initial linear portion of the load-extension curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. Before calculating, correct the extension for "toe compensation" as described in Annex A1. For purposes of this determination, the tensile stress shall be calculated by dividing the load by the average original cross section of the test section. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch), and reported to three significant figures.

11.8 *Secant Modulus*, at a designated strain, shall be calculated by dividing the corresponding stress (nominal) by the designated strain. Elastic modulus values are preferable and shall be calculated whenever possible. However, for materials where no proportionality is evident, the secant value shall be calculated. Draw the tangent as directed in A1.3 and Fig. A1.2 of Annex A1, and mark off the designated strain from the yield point where the tangent line goes through zero stress. The stress to be used in the calculation is then determined by dividing the load at the designated strain on the load-extension curve by the original average cross-sectional area of the specimen.

11.9 *Tensile Energy to Break*, where applicable, shall be calculated by integrating the energy per unit volume under the stress-strain curve or by integrating the total energy absorbed and dividing it by the volume of the original gage region of the specimen. As indicated in Annex A2, this may be done directly during the test by an electronic integrator, or subsequently by computation from the area of the plotted curve. The result shall be expressed in energy per unit volume, usually in megajoules per cubic metre (or inch-pounds-force per cubic inch). This value shall be reported to two significant figures.

11.10 For each series of tests, the arithmetic mean of all values obtained shall be calculated to the proper number of significant figures.

11.11 The standard deviation (estimated) shall be calculated as follows and reported to two significant figures:

$$s = \sqrt{(\sum X^2 - n\bar{X}^2)/(n - 1)}$$

where:

s = estimated standard deviation,
 X = value of a single observation,
 n = number of observations, and

\bar{X} = arithmetic mean of the set of observations.

12. Report

12.1 Report the following information:

- 12.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, and orientation of samples with respect to anisotropy (if any).
- 12.1.2 Method of preparing test specimens.
- 12.1.3 Thickness, width, and length of test specimens.
- 12.1.4 Number of specimens tested.
- 12.1.5 Strain rate employed.
- 12.1.6 Grip separation (initial).
- 12.1.7 Crosshead speed (rate of grip separation).
- 12.1.8 Gage length (if different from grip separation).
- 12.1.9 Type of grips used, including facing (if any).
- 12.1.10 Test method (A or B).
- 12.1.11 Conditioning procedure (test conditions, temperature, and relative humidity if nonstandard).
- 12.1.12 Anomalous behavior such as tear failure and failure at a grip.
- 12.1.13 Average breaking factor and standard deviation.
- 12.1.14 Average tensile strength (nominal) and standard deviation.
- 12.1.15 Average tensile strength at break (nominal) and standard deviation.
- 12.1.16 Average percent elongation at break and standard deviation.
- 12.1.17 Where applicable, average tensile energy to break and standard deviation.
- 12.1.18 In the case of materials exhibiting "yield" phenomenon: average yield strength and standard deviation; and average percent elongation at yield and standard deviation.
- 12.1.19 For materials which do not exhibit a yield point: average —% offset yield strength and standard deviation; and average percent elongation at —% offset yield strength and standard deviation.
- 12.1.20 Average modulus of elasticity and standard deviation (if secant modulus is used, so indicate and report strain at which calculated), and
- 12.1.21 When an extensometer is employed, so indicate.

13. Precision and Bias

13.1 Two interlaboratory tests have been run for these tensile properties. The first was run for modulus only, in 1977, in which randomly drawn samples of four thin (~0.025 mm (0.001-in.)) materials were tested with five specimens in each laboratory. Elastic (tangent) modulus measurements were made by six laboratories, and secant (1%) modulus measurements were taken by five laboratories. The relative precision obtained in this interlaboratory study is in Table 3.

13.1.1 In deriving the estimates in Table 3, statistical outliers were not removed, in keeping with Practice E 691.⁷

13.1.2 The within-lab standard deviation of a mean value, S_N , in each case was determined from the standard deviation, S_n , of the five individual specimens as follows: $S_N = S_n/(5)^{1/2}$. The S_N values were pooled among laboratories for a given

⁷ Supporting data are available from ASTM Headquarters. Request RR: D20-1084.

TABLE 3 Precision Data for Modulus

Material	Thickness, mils	Tangent Modulus				
		Average, 10 ³ psi	S_n , 10 ³ psi	$S_{R,500}$, 10 ³ psi	I_n , 10 ³ psi	$I_{R,500}$, 10 ³ psi
LDPE	1.4	53.9	1.81	8.81	5.12	24.9
HDPE	1.6	191	5.47	16.2	16.5	45.9
PP	1.1	425	10.3	31.5	29.0	89.1
PET	0.9	672	13.8	55.5	39.1	157.1

Secant Modulus						
LDPE	1.4	45.0	2.11	3.43	5.98	9.70
HDPE	1.6	150	3.29	9.58	9.30	27.1
PP	1.1	372	4.66	26.5	13.2	74.9
PET	0.9	640	10.0	27.5	28.4	77.8

material to obtain the within-lab standard deviation, S_n , of a test result (mean of five specimens). See 13.3 through 13.3.2 for definitions of terms in the tables.

13.2 An interlaboratory test was run for all the other tensile properties except modulus in 1981, in which randomly drawn samples of six materials (one of these in three thicknesses) ranging in thickness from 0.019 to 0.178 mm (0.00075 to 0.007 in.) were tested in seven laboratories. A test result was defined as the mean of five specimen determinations. However, each laboratory tested eight specimens, and the S_x was determined from $S_x = S_n(5)^{1/2}$ as above. This was done to improve the quality of the statistics while maintaining their applicability to a five-specimen test result. The materials and their thicknesses are identified in Tables 4 through 8, each of which contain data for one of the following properties: tensile yield stress, yield elongation, tensile strength, tensile elongation at break, and tensile energy at break (see Note 18).⁸

⁸ Supporting data are available from ASTM Headquarters. Request RR: D20-1101.

Note 18—Subsequent to filing the research report, examination of the LDPs used in this study between crossed polarizers revealed lengthwise lines representing substantial widthwise variation in molecular orientation that probably was not successfully randomized out of the between-labs component of variance.

Note 19—Caution: The following explanations of I_n and I_R (13.3 through 13.3.3) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table(s) 3 through 8 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.3 through 13.3.3 would then be valid for such data.

13.3 Concept of I_n and I_R —If S_n and S_R have been calculated from a large enough body of data, and for test results that were averages (medians/other function) from testing five specimens:

13.3.1 Repeatability, I_n (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the I_n value for that material.

13.3.2 Reproducibility, I_R (Comparing two test results for the same material, obtained by different operators using different equipment on different days)—The two test results should be judged not equivalent if they differ by more than the I_R value for that material.

13.3.3 Any judgment made in accordance with 13.3.1 and 13.3.2 would have an approximate 95 % (0.95) probability of being correct.

13.4 Bias—The systematic error which contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for these test methods.

14. Keywords

14.1 modulus of elasticity; plastic film; plastic sheeting; tensile properties; tensile strength; toughness; yield stress

TABLE 4. Precision Data for Yield Stress

Material	Thickness, mils	Average, 10^3 psi	$(S_y)^A$ 10^3 psi	$(S_y)^B$ 10^3 psi	$(S_y)^C$ 10^3 psi	$(S_y)^D$ 10^3 psi
LDPE	1.0	1.49	0.051	0.13	0.14	0.37
HDPE	1.0	4.33	0.084	0.16	0.24	0.44
PP	0.75	6.40	0.18	0.52	0.37	1.48
PC	4.0	8.59	0.072	0.29	0.20	0.82
CTA	5.3	11.4	0.12	0.50	0.34	1.43
PET	4.0	14.3	0.12	0.23	0.34	0.66
PET	2.5	14.4	0.14	0.54	0.40	1.52
PET	7.0	14.4	0.13	0.36	0.37	1.03

^A S_y is the within-laboratory standard deviation of the average.^B S_y is the between-laboratories standard deviation of the average.^C $t_1 = 2.83 S_y$ ^D $t_2 = 2.83 S_y$

TABLE 5. Precision Data for Yield Elongation

Material	Thickness, mils	Average, %	$(\epsilon_y)^A$ %	$(\epsilon_y)^B$ %	$(\epsilon_y)^C$ %	$(\epsilon_y)^D$ %
PP	0.75	3.5	0.15	0.41	0.42	1.2
PET	2.5	6.2	0.26	0.92	0.74	2.6
PET	4.0	5.3	0.25	0.60	0.71	1.7
PET	7.0	5.4	0.14	1.05	0.40	3.0
CTA	5.3	5.4	0.19	0.99	0.54	2.8
PC	4.0	6.9	0.24	0.98	0.68	2.8
HDPE	1.0	8.8	0.32	1.82	0.91	5.2
LDPE	1.0	10.0	0.55	3.41	1.56	9.6

NOTE—See Table 4 for footnote explanation.

TABLE 6. Precision Data for Tensile Strength

Material	Thickness, mils	Average, 10^3 psi	$(S_t)^A$ 10^3 psi	$(S_t)^B$ 10^3 psi	$(S_t)^C$ 10^3 psi	$(S_t)^D$ 10^3 psi
LDPE	1.0	3.42	0.14	0.53	0.40	1.5
HDPE	1.0	6.87	0.27	0.81	0.76	2.3
PC	4.0	12.0	0.34	0.93	0.96	2.6
CTA	5.3	14.6	0.20	1.37	0.57	3.9
PP	0.75	28.4	1.57	4.56	4.4	12.9
PET	4.0	28.9	0.65	1.27	1.8	3.6
PET	7.0	30.3	0.83	1.32	2.3	3.7
PET	2.5	30.6	1.22	2.64	3.4	7.5

NOTE—See Table 4 for footnote explanation.

TABLE 7. Precision Data for Elongation at Break

Material	Thickness, mils	Average, %	$(\epsilon_b)^A$ %	$(\epsilon_b)^B$ %	$(\epsilon_b)^C$ %	$(\epsilon_b)^D$ %
CTA	5.3	26.4	1.0	4.3	3	12
PP	0.75	57.8	4.4	12.7	12	36
PET	2.5	120	8.0	14.6	23	41
PET	7.0	132	5.8	10.8	16	30
PET	4.0	134	4.4	12.2	12	35
PC	4.0	155	5.4	17.1	15	48
LDPE	1.0	205	24.4	73.3	69	210
HDPE	1.0	570	26.0	91.7	74	260

NOTE—See Table 4 for footnote explanation.

TABLE 8. Precision Data for Tensile Energy to Break

Material	Thickness, mils	Average, 10^3 in./lb in. ³	$(S_e)^A$ 10^3 in./lb in. ³	$(S_e)^B$ 10^3 in./lb in. ³	$(S_e)^C$ 10^3 in./lb in. ³	$(S_e)^D$ 10^3 in./lb in. ³
CTA	5.0	3.14	0.14	0.70	0.4	2.0
LDPE	1.0	5.55	0.84	2.47	2.4	7.0
PP	0.75	11.3	1.19	3.11	3.4	8.8
PC	4.0	12.9	0.59	1.55	1.7	4.4
HDPE	1.0	26.0	1.87	5.02	5.3	14.2
PET	2.5	26.1	2.13	4.20	6.0	11.9
PET	4.0	27.1	1.42	2.75	4.0	7.8
PET	7.0	28.4	1.71	2.72	4.8	7.7

NOTE—See Table 4 for footnote explanation.



ANNEXES

(Mandatory Information)

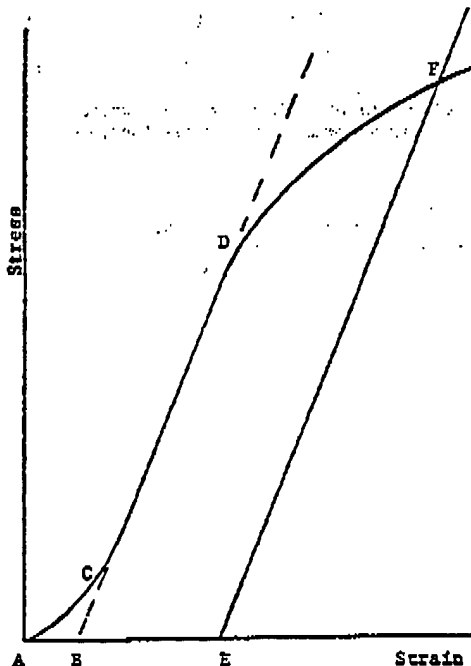
A1: TOE COMPENSATION

A1.1 In a typical stress-strain curve (Fig. A1.1) there is a toe region, AC , which does not represent a property of the material. It is an artifact caused by a takeup of slack, and alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus, strain, and offset yield point, this artifact must be compensated for to give the corrected zero point on the strain or extension axis.

A1.2 In the case of a material exhibiting a region of Hookean (linear) behavior (Fig. A1.1), a continuation of the linear (CD) region of the curve is constructed through the zero-stress axis. This intersection (B) is the corrected zero-strain point from which all extensions or strains must be

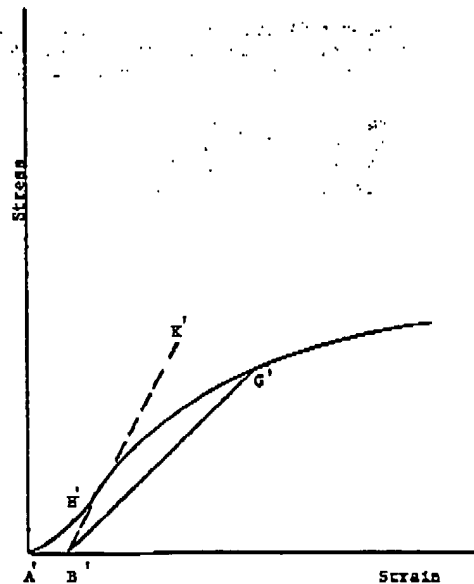
measured, including the yield offset (BE), if applicable. The elastic modulus can be determined by dividing the stress at any point along the line CD (or its extension) by the strain at the same point (measured from point B , defined as zero-strain).

A1.3 In the case of a material that does not exhibit any linear region (Fig. A1.2), the same kind of toe correction of the zero-strain point can be made by constructing a tangent to the maximum slope at the inflection point (H). This is extended to intersect the strain axis at point B' , the corrected zero-strain point. Using point B' as zero-strain, the stress at any point (G') on the curve can be divided by the strain at that point to obtain a secant modulus (slope of line $B'G'$). For those materials with no linear region, any attempt to use the tangent through the inflection point as a basis for determination of an offset yield point may result in unacceptable error.



NOTE—Some chart recorders plot the mirror image of this graph.

FIG. A1.1 Material with Hookean Region



NOTE—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

TABLE 4 Precision Data for Tensile Test

A2. DETERMINATION OF TENSILE ENERGY TO BREAK

A2.1 Tensile energy to break (TEB) is defined by the area under the stress-strain curve, or

$$TEB = \int_0^{\epsilon_r} S d\epsilon$$

where S is the stress at any strain, ϵ , and ϵ_r is the strain at rupture. The value is in units of energy per unit volume of the specimen's initial gage region. TEB is most conveniently and accurately measured with a tension tester equipped with an integrator. The calculation is then:

$$TEB = (I/K) \frac{(\text{full scale load}) (\text{chart speed}) (\text{crosshead speed/chart speed})}{(\text{mean caliper}) (\text{specimen width}) (\text{gage length})}$$

where I is the integrator count reading and K is the maximum possible count per unit time for a constant full scale load. This whole calculation is typically done electronically. The results are best expressed in megajoules per cubic metre (or inch-pounds-force per cubic inch).

A2.2 Without an integrator, the area under the recorded stress-strain curve can be measured by planimeter, counting

squares, or weighing the cut-out curve. These techniques are time-consuming and likely to be less accurate, since the load scale on some chart paper is not in round-number dimensions. Moreover, if the curve coordinates are in terms of force and extension instead of stress and strain, the calculated energy, corresponding to the measured area, must be divided by the product of gage length, specimen width, and mean caliper:

$$TEB = \frac{(\text{curve area}) (\text{force per unit chart scale})}{(\text{extension per unit chart travel}) (\text{mean caliper}) (\text{specimen width}) (\text{gage length})}$$

A2.3 For example, if the area under a force-extension curve is 60 000 mm², the load coordinate is 2.0 N/mm of chart scale, the extension coordinate is 0.25 mm of extension per mm of chart travel, and the specimen dimensions are 0.1 mm caliper, 15 mm width and 100 mm gage length, then the calculation for tensile energy to break is:

$$TEB = \frac{(60\,000\text{ mm}^2) (2.0\text{ N/mm}) (0.25 \times 10^{-3}\text{ m/mm})}{(0.1 \times 10^{-3}\text{ m}) (15 \times 10^{-3}\text{ m}) (100 \times 10^{-3}\text{ m})}$$

$$TEB = 200\text{ MJ/m}^3$$

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